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(58) Field of Search

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(54) Abstract Title

**A coated particulate aggregate material for use in construction**

(57) A particulate material, suitable for use in the construction of road and airfield surfaces, comprises aggregate particles coated with a set mixture of cement and water, wherein the particles may comprise crushed or uncrushed aggregate, recycled aggregate or artificial aggregate. The coated particles are rougher than the uncoated aggregates. Preferably, natural uncrushed aggregate, e.g. gravels which are smooth and rounded, are coated. Extra lime can be added to the cement, as can microsilicas, setting and/or hardening accelerators, and/or superplasticizers.

Additional smaller particulate matter can be included in the coating, some particles of which project from the surface of the coating.

The particulate material is made by mixing the aggregate particles with cement, water and any additives, separating the particles by passing over or through vibrating/rotating sieves before the cement sets, and then conveying the separated particles on vibrating conveyor(s) until the cement substantially reaches its initial setting.

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At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

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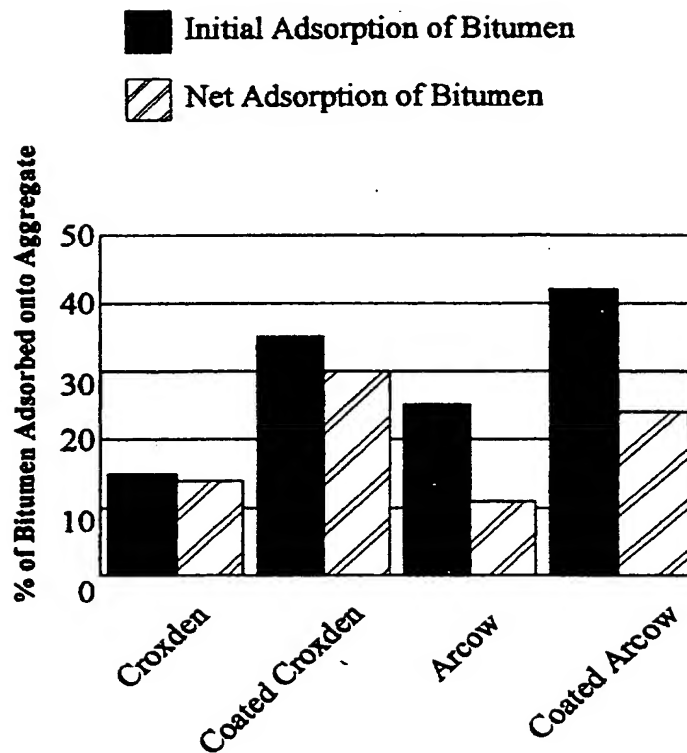


Fig. 2.

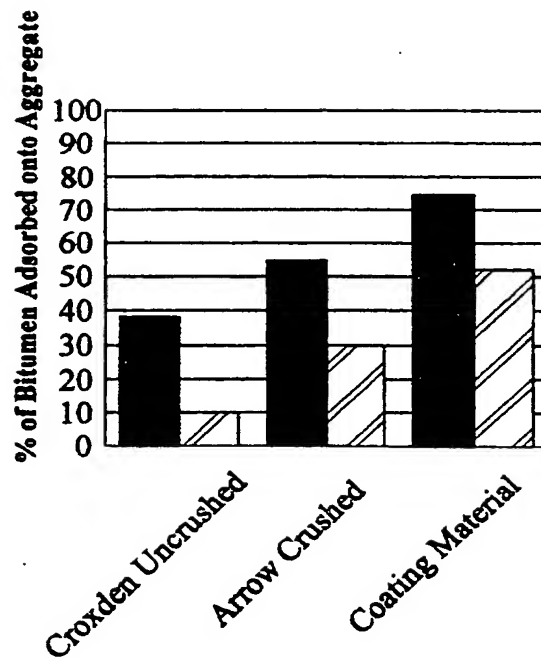
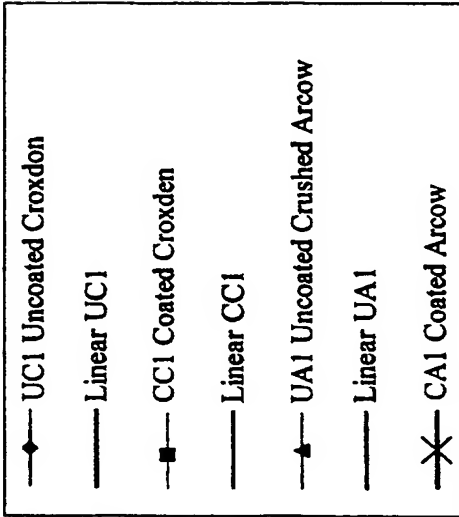


Fig. 3.

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Aggregate	Angle of friction (Interlocking resistance)	Improvement % in interlocking
Uncoated Croxdon UCI	11.9°	47
Coated Croxdon CCI	17.48°	
Uncoated Crushed Arcow UAI	14.42°	21.2
Coated Arcow CAI	17.48°	

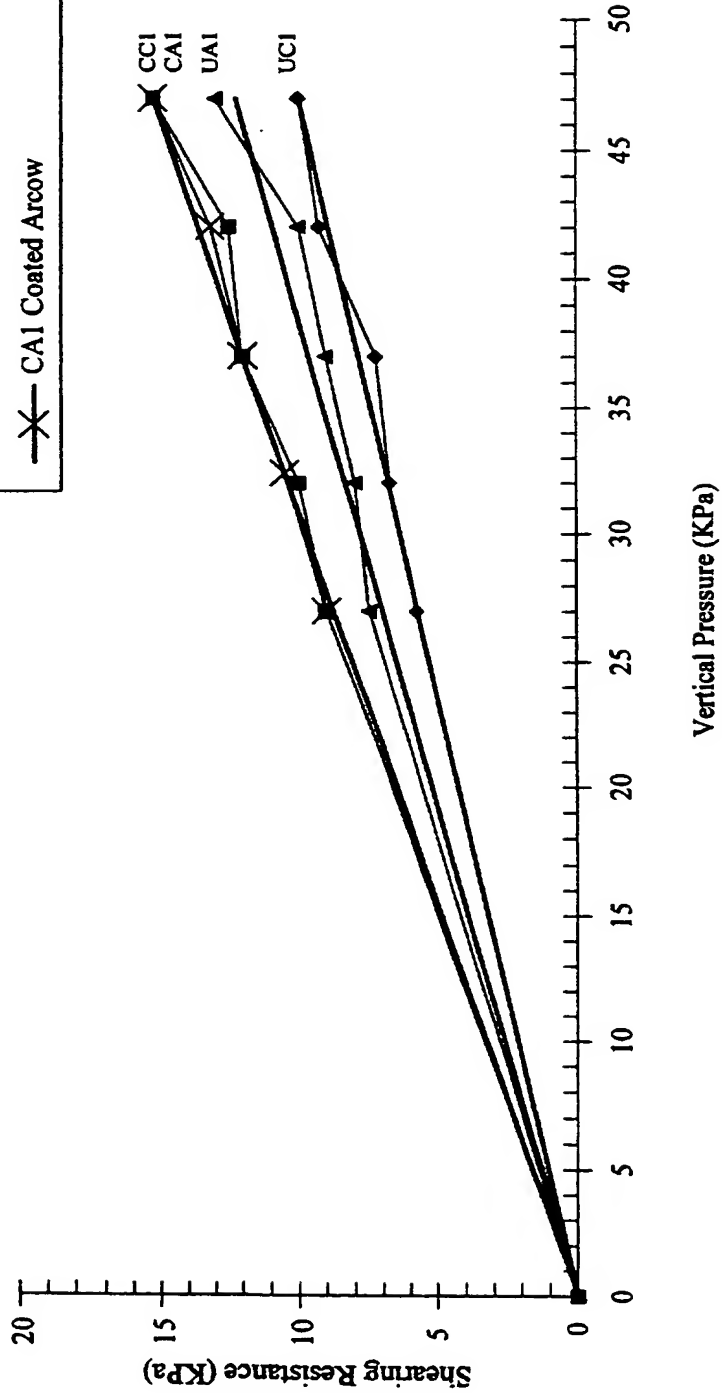


Fig. 4.

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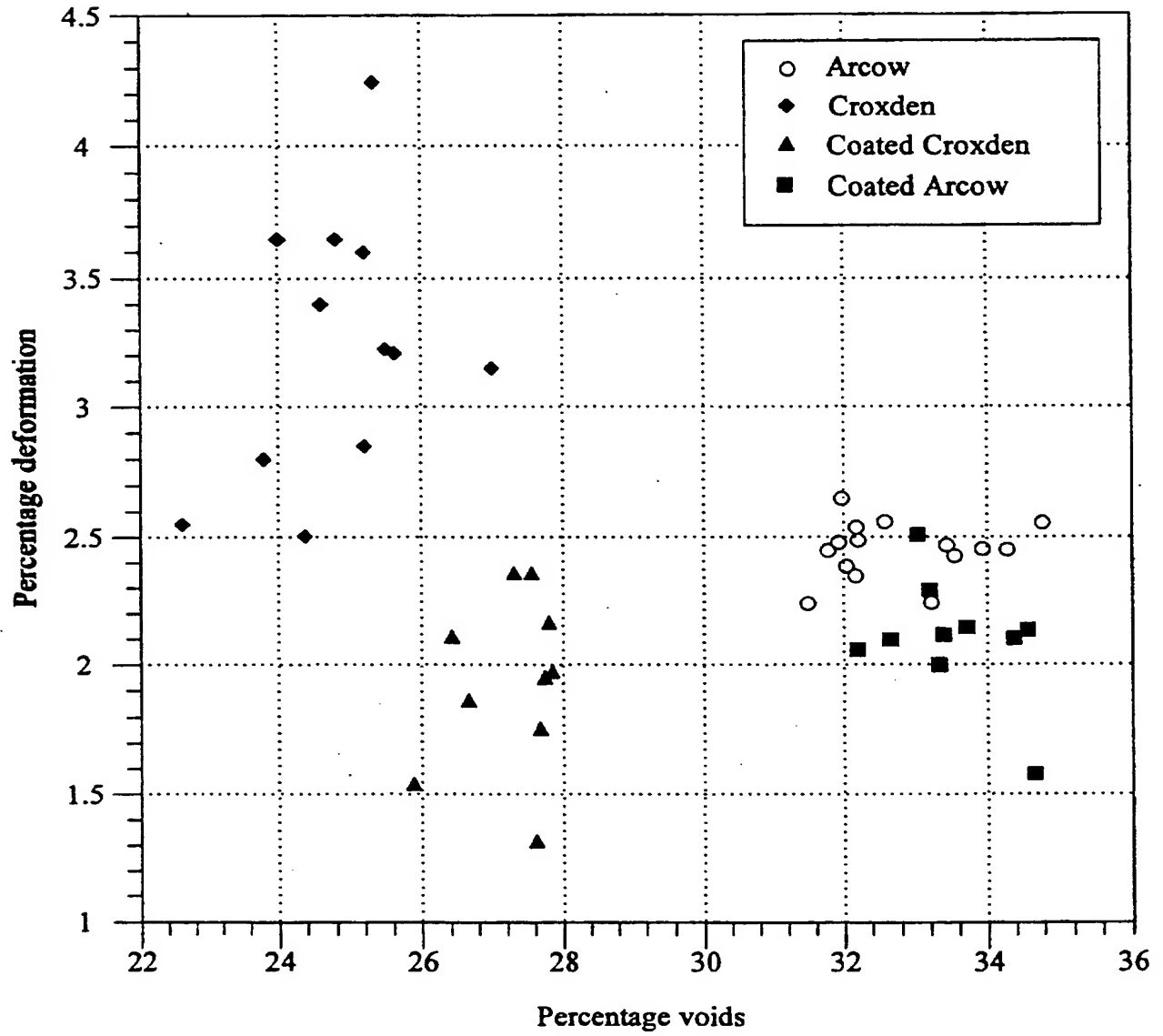


Fig. 5.

DESCRIPTIONA PARTICULATE MATERIAL SUITABLE FOR USE IN  
CONSTRUCTION AND METHODS OF MAKING SAME

The present invention relates to a particulate material comprising modified aggregate particles suitable for, for example, the formation of surfaces (e.g. the surfacing layers or roadbase of flexible pavement structures) for city streets, highways, airfields and other construction applications, and is concerned with a method of modifying such aggregates to improve their characteristics and usefulness in these fields.

The current practice in the pavement construction industry is to use crushed rock aggregates of "suitable" characteristics. Although natural uncrushed rock aggregate, recycled aggregate and conventionally "unsuitable" crushed rock aggregate exist in large quantities and there is a huge demand for their use in the construction industries, they are not used currently in pavement construction because of the inability at present to produce reasonable quality pavement from these materials because of their inherent nature. For example, the surface of most natural uncrushed aggregate (e.g. from riverbeds, seabeds, deserts etc) is too smooth for pavement construction purposes, as described below.

Figures from the British Aggregate Construction Materials Industries (BACMI) (Statistical Yearbook 1994, BACMI, London, 1994) show that in a sample period, 27.4 million tonnes of crushed rock, compared to only 0.6 million tonnes of uncrushed gravel, was used in road construction.

Furthermore, it is being realised that the environmental impact of development within the community has reached an unacceptable high. In particular, the crushed rock aggregates presently used in pavement construction have a negative environmental impact in that their production involves quarrying, i.e. dynamiting mountains and crushing the resultant broken rock into the desired particle size. Consequently, there has been an increased desire to produce and repair roads with longer lasting, more environmentally friendly materials. In particular, an environmental benefit could be achieved if it were possible to make use of (presently unsuitable) natural aggregates from river beds and deserts etc. However, these materials are still required to deliver a comfortable, safe ride under increasingly severe conditions.

Throughout the transportation industry, there is therefore an ongoing need to improve the quality of the materials provided for the construction of flexible pavements. Flexible bituminous pavements constitute the largest part of the transportation network facility in many countries. Therefore, the performance and durability of bituminous material requires continual upgrading to meet the socio-economic and environmental demands of the transportation network user.

Some of the problems associated with these currently unusable aggregate materials, in particular natural gravels, are now discussed.

**1) Bituminous mixes containing natural uncrushed aggregate quickly age and become brittle**

**1.1. A report by Griffith and Kallas (Aggregate Void Characteristics in**

Asphalt Paving Mixes, Griffith, J.M. and Kallas, B.F. Proceedings of the HRB, Vol. 36, 1957) stated that mixtures containing natural aggregate would generally require less asphalt than those containing crushed stone aggregate. This was attributed to the fact that natural gravels develop lower aggregate voids when compared to crushed stone mixes having the same gradation. This property, although appearing beneficial, would indicate a tendency for uncrushed natural aggregate asphalts to deform easily under the action of the traffic loads and in the presence of bituminous binder which has to be used at a specified optimum content. In porous mixes and other like mixes, the tendency for an aggregate to require less binder is not desirable as the finished product will quickly age and become brittle. That will reduce the design life of the pavement layers.

1.2. A study by Curtis et al. (Fundamental Properties of Asphalt-Aggregate Interactions Including Adhesion and Adsorption, Strategic Highways Research Program (SHRP), Report A-341, National Research Council, Washington DC, Main Contributors: Christine W. Curtis - NCAT/Auburn University, Keith Ensely - Western Research Institute, Jon Epps - University of Nevada at Reno, 1993) showed that when asphalt was desorbed from a selection of Materials Reference Library (MRL) aggregates, desorption of asphalt occurred in the following order. Gravels > Limestones > Greywackes > Granites. Gravel asphalts appear therefore to be the most vulnerable to attack from the ingress of water into the mix. This indicates that the bond between asphalt and either round gravel aggregates or gravel with a



polished surface is poor compared to the bond between crushed limestone or granite aggregates and asphalts.

1.3. A further study by Curtis et al. ("Asphalt/Aggregate Mix Susceptibility Test Based on Chemical and Physical Bond Strength") analysed the Tensile Stress Ratio (TSR) of various MRL aggregates with the Modified Lottman Test. The two gravel aggregates exhibited some of the lowest TSR values of all of the aggregates tested, indicating a poor chemical and physical bond.

**2) Bituminous pavement containing natural uncrushed aggregate lacks stability and leads to rutting**

2.1. Kandal, Khatri and Motter (Evaluation of Particle Shape and Texture of Mineral Aggregates and Their Blends, Kandhal, P.S. Khatri, M.A., Motter, J.B. NCAT Report No. 92-4 Presented at the Annual Meeting of the Association of Asphalt Paving Technologists (AAPT) in Charleston, SC, 1992) showed that using BB (Lead shot, in both its natural rounded shape, and in a flattened form), to simulate natural gravel is very undesirable in asphalt mixes. This is because there is no interlocking of the particles that produce stability. This lack of stability leads to visco-elastic flow, more simply referred to as rutting.

2.2. Wedding and Gaynor (The Effects of Using Crushed Gravel as the Course and Fine Aggregate in Dense Graded Bituminous Mixes, Wedding, P.A. and Gaynor, R.D. Proceedings of the AAPT, Vol. 30, 1961) reported that when crushed gravel was used in place of uncrushed gravel in an HMA

(Hot Mix, or Hot Rolled Asphalt) mixture, an increase in stability of approximately 45 percent could be achieved with the substitution of all crushed aggregate (crushed gravel course and fine aggregate) by natural sand and gravel.

2.3 Field (The Importance of Percent Crushed in Course Aggregate as Applied to Bituminous Pavements, Field, F. Proceedings of the AAPT, Vol. 27, 1958) studied the influence of the percentage of the crushed particles of the course aggregate in HMA mixtures. He reported that Marshall stability of an HMA mix changed little for 0 to 35 percent crushed particles and then increased consistently as the percentage of crushed particles increased to 100 percent. The average stability was reported to be 55 percent higher for 100 percent crushed particles as compared to that of 35 percent crushed particles.

2.4 Campen and Smith (A Study of the Role of Angular Aggregates in the Development of Stability of Bituminous Mixes, Campen, W.H. and Smith, J.R. Proceedings of the AAPT, Vol. 17, 1948) have reported an increase in stability of 30 to 190 percent by using crushed aggregate as compared to natural rounded aggregate in HMA mixtures. They measured the stability by Hubbard-Field and Bearing-Index tests.

3. A paper has been published on research in hot climates to mix crushed aggregate with cement powder but without any added water for use in asphalt mixes. The material is limited to crushed rock aggregate only, however.

4. Finally, a paper by R.J. Schmidt and P.E. Graf (The Effect of

Water on the Resilient Modulus of Asphalt-treated Mixes, Proceedings of the AAPT, Vol. 41, 1972, p.p. 118-162) describes a process in which conventionally suitable crushed aggregates are dipped in a slurry of lime. The product was left for 24hr after dipping and then used in an asphaltic mix to increase the adhesion of binder to the crushed aggregates.

It would be desirable if a way could be found to modify aggregates of types not currently considered useable, especially natural uncrushed aggregate, so that they could be used satisfactorily for construction purposes, in particular construction of surfaces (e.g. flexible pavement).

According to a first aspect of the present invention there is provided a particulate material suitable for use in construction comprising aggregate particles having a coating, wherein the coating comprises a set mixture of cement and water, and wherein the aggregate particles comprise natural uncrushed aggregate, recycled aggregate or artificial aggregate.

Alternatively, in the present invention the aggregate particles can comprise crushed aggregate which, when uncoated, is unsuitable for use in construction because of its physical, mechanical and chemical properties, for example because the aggregate surface is not strong enough, has a plurality of voids, or has a high surface polishing value such that the aggregate surface polishes and becomes smooth too quickly during use. Providing the cement coating can make these aggregates suitable for use in construction of, for example, flexible pavement.

In the context of the present invention, the term "cement" includes

standard mineral-based powders used for construction purposes (e.g. as used in concrete, mortar, etc) which when mixed with water set to a hard mass.

Preferably, the cement comprises one or more calcium compounds such as lime. More preferably, the cement comprises calcium, silicon and aluminium in the form of one or more compounds.

Even more preferably, the cement comprises calcium, silicon, aluminium and oxygen in the form of one or more oxygen-containing compounds each containing one or more of calcium, silicon and aluminium (e.g. lime, silica  $\text{SiO}_2$ , alumina  $\text{Al}_2\text{O}_3$ , silicates, aluminates, aluminosilicates, etc.). The cement can also comprise iron, magnesium and/or sulphur in the form of one or more oxygen-containing compounds each containing one or more of iron (e.g.  $\text{Fe}_2\text{O}_3$ , aluminoferrates), magnesium (e.g.  $\text{MgO}$ ) and sulphur (e.g. calcium sulphate), which can be separate oxygen-containing compounds and/or the same as the one or more oxygen-containing compounds each containing one or more of calcium, silicon and aluminium. One example is Portland cement. More details about the constituents of cement can be found in F.M. Lea, The Chemistry of Cement and Concrete, Third Edition, Edward Arnold Publishers.

"Lime" is hereby defined as  $\text{CaO}$  and/or the nominal  $\text{CaO}$  content of other calcium compounds, i.e. excluding the nominal content of alumina, silica, sulphur trioxide and iron oxide, etc. within the calcium compounds (e.g. aluminates, silicates, sulphates, etc.) present. "Limes" is construed in the same way as "lime".

References to "set" or "setting" include the initial setting process of the cement coating.

"Natural uncrushed aggregate" means aggregate obtained from natural resources (e.g. deserts, riverbeds, seabeds, etc.) which is naturally present in aggregate form and which has not been crushed to size by man-made machinery. One example is natural gravel.

"Recycled aggregate" has the normal meaning understood by a person skilled in the art and can include aggregate used previously on roads or similar surfaces. When the old road surface is removed, the bitumen binder can be removed, and the remaining aggregate reused. This is not commonly reused for road surfaces.

"Artificial aggregate" means aggregate manufactured or synthesised by man, and usually comprises biproduct material from various industrial processes. One example is slag.

The present invention is advantageous in that providing aggregate particles with a cement coating enables aggregate particles of low quality, especially natural uncrushed aggregate but also artificial, recycled and unsuitable crushed aggregates, to be brought up to a recognised standard for use in pavement construction by means of a cheap and simple coating process. It also enables aggregate already of an acceptable quality (e.g. most crushed aggregate) to have its performance enhanced.

In the case of natural uncrushed aggregate, there has long been a need to use this cheap and environmentally friendly material in road construction.

However most skilled workers have not thought to use natural uncrushed aggregate in road construction as, when untreated, it has such poor qualities; past research has concentrated on conventionally suitable crushed aggregate. The present invention provides an unexpected method of making, in particular, natural uncrushed aggregate useable.

A first advantageous effect of the provision of the coating of cement (which contains lime) is to alter the characteristics of the aggregate surface from being hydrophilic or oleophobic (oil hating) to being a more oleophilic (oil loving) surface which has properties similar to those of a lime-rich aggregate. Lime rich or basic (alkalinic) uncoated aggregates are the least oleophobic and hence are the most likely to bond well with bitumen, as explained in the study of Curtis et al. referred to in ref 1.2. above. In the same way, the cement coated aggregate also generally forms a better bond with bitumen and/or asphalt binder and enables increased percentages of binder to be retained (adsorbed) on the coated aggregate (results are shown hereinafter).

The foregoing advantage results in that aggregate asphalt mixes containing the new coated aggregates generally require more binder than unacceptable (e.g. natural uncrushed) aggregate, and the finished product therefore generally ages less quickly, loses bitumen less quickly and is less liable to become brittle with time, ie. the resulting product generally has a long design life for flexible and composite pavements for city streets, highways, roads and airfields.

The fact that mixtures containing cement coated natural aggregate would generally require more asphalt binder than those containing uncoated natural or unsuitable crushed stone aggregate is attributed to the fact that natural gravels develop lower aggregate voids when compared to suitable crushed stone mixes having the same gradation. This property, although appearing beneficial, would indicate a tendency for uncrushed natural aggregate asphalts to deform easily if too much binder is included in the mix. Porous mixes are designed with the maximum amount of bitumen retained without binder drainage. Therefore in these mixes, as in those above, the tendency for an aggregate to require less binder is not desirable as the finished product would quickly age and become brittle.

Furthermore, a second advantage of the cement coating is that the surface of the aggregate generally becomes rougher (as described hereinafter), particularly when the coating contains small additional hard particles. This is a particular advantage for natural gravels which are smooth and rounded in their natural state. This increased roughness enables a greater degree of mechanical interlocking between adjacent aggregate particles, thereby resulting in increased shear resistance, which results in improved durability and stability since the phenomenon of visco-elastic flow under the action of the traffic loads is substantially reduced. Thus, the problem of "rutting" of pavement surfaces, which exists in particular with natural gravels which are presently unsuitable, can be substantially reduced or even eliminated, when the coated aggregates are used. Therefore the simple expedient of coating natural gravel renders it

useable for this application.

A third advantage of the present invention is that natural uncrushed and recycled aggregates are generally recovered with less energy intensive means than quarrying, which produces crushed aggregate, and could therefore be considered more environmentally-friendly. It is envisaged that the long term effect of using coated uncrushed and recycled materials of the present invention will be to reduce the cost and environmental impact of producing and maintaining roads.

Preferably, the aggregate particles comprise rock.

Preferably, the natural uncrushed aggregate comprises natural gravel.

Preferably, the particulate material includes coated particles containing only one aggregate particle. This material is the preferable but non-essential result of separating the coated particles before drying (initial setting) and thereby reducing the amount of "lumps" with more than one aggregate particle in.

Any particle size of aggregate can be used, but preferably the aggregate particles include those with a particle size of  $\geq 2.36$  mm (excluding the coating). More preferably, in order to create "coarse aggregate" coated material within the meaning of British Standard BS 4987 Part 1 (1993) for pavement construction use, the aggregate particles include those with a particle size of 2.36-50 mm (excluding the coating).

Even more preferably the aggregate particles include those with a particle size of 2.36-14 mm (excluding the coating). However, other particle



size ranges can be used before coating, for example 2.36-6.3 mm, 2.36-20 mm, 3.35-6.3 mm, 6.3-14mm, 14-28 mm, 28-37.5 mm and/or 37.5-50mm. In all cases, the particle sizes are measured using the appropriately sized sieves.

Preferably, the final particulate material (after coating) should include that with a particle size of  $\geq 3.35$  mm. More preferably, it should include that with a particle size of 3.35-50 mm, for example 3.35-6.3 mm, 6.3-14 mm, 14-28 mm, 28-37.5 mm, and/or 37.5-50 mm. Other particle sizes can also be included, however, e.g. 75-300  $\mu\text{m}$  and/or 300  $\mu\text{m}$ -3.35 mm.

Alternatively, the proportion of particles outside the given ranges can be reduced by careful sieving so that the aggregate particles (before coating) or the particulate material (after coating) substantially consist of those with the above particle sizes. For example, to create material within both the "coarse aggregate" (3.35-50 mm) and "sub-base" ( $> 50$  mm) categories of BS 4987, Part 1, 1993 for road use, the final particulate material after coating should substantially consist of that with a particle size of  $\geq 3.35$  mm. To create "coarse aggregate" only within the meaning of BS 4987, the final particulate material should substantially consist of that with a particle size of 3.35-50 mm.

Ideally, the coating substantially completely surrounds the aggregate particles. Usually, the coating has an average thickness of 0.1-6 mm, more usually of 0.5-3 mm.

It is advantageous to include various additives in the coating in addition to the cement, such as limes, microsilicas, and/or pulverised fuel ash (PFA). "Limes" have been defined previously. "Microsilicas" means condensed silica

fumes or dust, usually a very fine dust, (e.g. as formed in a synthetic chemical process) and are not normally present in cement. Microsilicas can react substantially completely within the set cement coating, in contrast to silica which only partly reacts, and therefore they increase the strength of the coating to a much greater extent than the silica found in normal cement.

In order to maximise the oleophilicity and bitumen bonding properties of the cement coating, the coating preferably has a total lime content of  $\geq 64$  wt % (more preferably 64-67 wt %) of the total dry weight of the cement and additives in the coating. Portland cement already contains lime (about 64.0-64.6 wt %, according to F.M. Lea, The Chemistry of Cement and Concrete, Edward Arnold Publishers, 3rd edition, page 16), and so it can be seen that additional lime may be advantageous depending on the lime content of the cement used and the end use of the final product. Preferably, Portland cement (e.g. Ordinary Portland Cement, OPC) or rapid-hardening cement will be used.

Preferably, the coating comprises a setting accelerator, a superplasticizer and/or a hardening accelerator. The function of the setting accelerator is to reduce the initial setting time of the cement coating, and thereby to minimise the time during which the coated aggregate particles need to be kept separate. Preferably, the setting accelerator comprises a mixture of organic and inorganic complex-forming agents. The superplasticizer promotes better cement dispersion. The hardening accelerator promotes improved high early strengths in (for example) the 8-24 hr period after coating.

In a highly advantageous embodiment the coating contains additional particulate matter.

More preferably, the additional particulate matter has a smaller average particle size than that of the aggregate particles, and/or the additional particulate matter includes that with a particle size of  $75\ \mu\text{m}$  -  $2.36\ \text{mm}$  or  $75\ \mu\text{m}$  -  $3.35\ \text{mm}$  (e.g.  $75\text{-}300\ \mu\text{m}$  and/or  $300\ \mu\text{m}\text{-}3.35\ \text{mm}$ ), and/or the additional particulate matter comprises hard angular particles. The ideal additional particulate matter is sand. Generally, a plurality of these small particles surround the central larger aggregate particle.

This additional small particulate matter can be encapsulated within and/or project from the surface of the coating. The projecting particles greatly increase the roughness and angularity of the final particulate material - generally to an average roughness of  $\geq 400\ \mu\text{m}$ , as measured by optical surface profile. In this way, the mechanical interlocking and shear resistance properties of the final material are improved further, which further increases the stability and decreases the visco-elastic flow under load of pavement and/or asphalt mixes made with this material. The encapsulated particulate matter serves to increase the strength of the coating.

In comparison, the particulate material (without additional small particulate matter) generally has an average roughness of  $\geq 100\ \mu\text{m}$ , more usually  $125\text{-}400\ \mu\text{m}$ , as measured by optical surface profile. Typically, the average roughness for coated natural uncrushed aggregate is  $125\text{-}200\ \mu\text{m}$  or  $125\text{-}180\ \mu\text{m}$ , and for coated crushed aggregate is  $200\text{-}400\ \mu\text{m}$ . This represents

a considerable improvement for both natural uncrushed and crushed aggregate, as detailed more fully in the "Test Results" section in Table 1 hereinafter.

According to a second aspect of the present invention there is provided a method of making a particulate material as hereinbefore described, comprising providing aggregate particles with a coating of cement paste, and allowing the cement coating to reach its initial setting.

Preferably, the method comprises mixing the aggregate particles with cement and water, and more preferably also comprises mixing the aggregate particles with additional limes.

Preferably, 1-12 wt. % of additional limes by weight of dry cement powder used are added, or more preferably 1-7 wt. % of additional limes by weight of dry cement powder used are added. Up to 3 wt. % of additional limes may be added for use in road surfacing.

The function of the additional limes is to increase the oleophilicity and bitumen-binding properties of the final particulate material, as described above. The percentages quoted are calculated with this function in mind.

Preferably, the method also comprises mixing the aggregate particles with microsilicas and/or pulverised fuel ash (PFA); usually 1-12 wt. % of microsilicas and/or PFA by weight of dry cement powder used are added. The microsilicas added are in addition to any microsilicas which might already be present in cement. The method can also comprise mixing the aggregate particles with a setting accelerator, a superplasticizer and/or a hardening accelerator as defined hereinabove.

A highly advantageous embodiment of the method is to mix the aggregate particles with the additional particulate matter as described hereinabove and for the same purposes.

Ideally, the coated aggregate particles are separated before the cement coating is allowed substantially to reach its initial setting; more specifically the coated aggregate particles can be separated by passing through or over one or more rotating and/or vibrating sieves.

This stage helps to make the final coated material particulate, as opposed to a large solid block or mass of set concrete. Use of multiple sieves can divide the coated product into different size fractions if desired.

Preferably, the method comprises disposing the coated aggregate particles onto a conveyor before the cement is allowed substantially to reach its initial setting. Usually, the conveyor vibrates in such a way as to aid separation of the coated aggregate particles from adjacent coated aggregate particles. This last feature helps to reduce formation of "lumps" containing more than one larger aggregate particle (e.g. of  $\geq 3.35$  mm uncoated size). Usually, the coated aggregate particles are retained on the conveyor and/or an equivalent mechanism for a time sufficient to allow the coating to reach its initial setting at least partially. This reduces "lump" formation when the particulate material is deposited in a hopper.

Preferably, dry powder, for example dry cement and/or accelerating powder, is added to the coated aggregate particles while they are still on the conveyor. When this dry powder contacts the wet coated aggregate particles,

it is partially retained in the cement paste and helps to accelerate the initial setting of the cement.

Sometimes, the coated aggregate particles may be passed through or over one or more sieves after conveying. This allows a final grading of the product, possibly into different size fractions and again helps to remove lumps.

According to a third aspect of the present invention there is provided a particulate material made by a method as hereinbefore described.

According to a fourth aspect of the present invention there is provided a method of using a particulate material of the present invention, comprising mixing said particulate material with an asphalt and/or bitumen binder. This mixing would normally occur at the roadside or on plant.

According to a fifth aspect of the present invention there is provided a material suitable for use in construction, comprising an asphalt and/or bitumen binder and a particulate material of the present invention.

According to a sixth aspect of the present invention there is provided the use in construction of a particulate material of the present invention or a material of the fifth aspect of the present invention.

A preferable use is in the construction of surfaces (e.g. flexible pavement), such as road or airfield surfaces. "Surface" in this context means a layer or layers of defined thickness and is not to be construed as meaning only the outer boundary of that layer or layers. A particulate material or material of the present invention can be used as a substitute for conventional aggregate within the body of a road surface, for example within the "surfacing layers"

(comprising the wearing course and base course) or "roadbase" layer of pavement structures.

The particulate material or material of the present invention, with or without a coating of asphalt or other suitable material, can also be used as "chippings" or for surface dressing. Chippings are the last aggregate materials compressed into the road surface on top of the top asphalt-containing layer of the pavement structure. Use of rough cement coated materials of the present invention as chippings may give the road surface improved friction/skid resistance properties as well as the normal advantages.

According to a seventh aspect of the present invention there is provided a method of constructing a surface comprising applying a material of the fifth aspect of the present invention to another surface.

According to an eighth aspect of the present invention there is provided a surface containing a particulate material of the present invention or a material of the fifth aspect of the present invention.

According to a ninth aspect of the present invention there is provided an aggregate material of natural, artificial or crushed rock particles having a cement coating. Preferable embodiments of this aggregate material are as for a particulate material of the first aspect of the present invention.

According to a tenth aspect of the present invention there is provided a method of modifying aggregate materials in order to improve their characteristics for use in pavement construction, wherein the aggregate material is provided with a coating of cement paste. Preferable embodiments

are as for a method of making a particulate material of the second aspect of the present invention. Pavement preferably includes road pavement or airfield pavement.

It is noted that the materials and/or methods of the present invention may find a use outside the construction field and the scope of the present invention should not be construed so as to exclude these applications.

### EXAMPLES

A specific embodiment of the invention will now be described, by way of example only, and by reference to the drawings attached hereto, in which:

Fig. 1a, represents a particle (2) of one embodiment of the present invention, comprising an aggregate particle (4) having a cement coating (6); and

Fig. 1b shows particle (8) of another embodiment of the present invention, comprising an aggregate particle (10) having a cement coating (12) containing additional particulate matter (14,16) some of which (16) is encapsulated within the coating and some of which (14) projects from the surface (18) of the coating (12).

Figs 2-4 are described later in the "Test Results" section.

The coated aggregate is made as follows.

In brief, the cement coating is preferably applied to the aggregates by mixing the latter with the cement and water, with or without additives, in a conventional concrete mixer or any other suitable mixer. The quantities of the materials should be in the amounts specified below. After mixing, the coated



aggregates are separated by passing through a gently vibrated sieve or other suitable mechanism and/or dropping onto a vibrating conveyor. After the initial setting time of the cement, conventional conveying techniques are applied and the material will be ready for use.

Details of the method of making the coated aggregate are as follows. Natural uncrushed or crushed aggregate are separated into fractions with particle sizes of 2.36-6.3 mm or 3.35 - 6.3 mm, 6.3 - 14 mm, 14-28 mm, 28-37.5 mm and 37.5-50 mm using sieves of the given mesh size. Alternatively, fractions with a larger particle size range (e.g. 2.36-14 mm or 2.36-20 mm, as measured by the appropriate sieves and as used in the following tests) can be used. Each fraction of aggregate is mixed with dry cement powder, any additives and water in a conventional concrete mixer, for the standard time as in the standard concrete-mixing process.

The proportions of aggregate, cement, additives and water are as follows. 3-20 wt. % cement by weight of dry aggregate is used - either ordinary Portland cement (OPC), which is cheapest, or rapid-hardening cement (e.g. cement with a high alumina and/or microsilica content). In this specific embodiment, 10 wt. % of OPC by weight of dry aggregate is used, in which case water is added according to a water: cement ratio of 4.2% (or as according to the relevant International Standard), adjustable to the proportion of the constituents of the product.

1-12 wt. % of microsilicas and/or PFA, by weight of dry cement used, may be used as an additive.

A cement and additives mix with a combined lime content of at least 64 wt%, or alternatively 64-67wt.%, should be used in order to increase the oleophilicity of the final coated product and improve bonding to bitumen. "Lime" has been defined previously. OPC has a lime content of about 64.0-64.6% (see before). Therefore, 1-12 wt.% or 1-7 wt % of extra lime by weight of dry cement is added to the OPC, in order to increase the oleophilicity and bitumen-bonding properties of the cement coating. Alternatively, the amount of added lime may be as small as 0-3 wt.% for use on roads, but may be more for other applications.

In an alternative embodiment additional fine aggregate, e.g. sand, with a particle size of  $75\ \mu\text{m}$  - 2.36 mm or  $75\ \mu\text{m}$  - 3.35 mm (e.g.  $300\ \mu\text{m}$  - 3.35 mm and/or 75 -  $300\ \mu\text{m}$ ) is also added to the mix in various proportions to increase the roughness and interlocking characteristics of the final coated aggregate (see later). This additional fine particulate matter (14,16) can be encapsulated within (16) or project from (14) the surface (18) of the final dried coating (12), as shown in Fig. 1b.

In an advantageous embodiment, a setting accelerator additive is used in the mixture to decrease the setting time of the cement. A setting accelerator containing organic and inorganic complex-forming agents such as Sika®Rapid-2, produced by Sika AG, Tüffenwies 16, 8048 Zürich, Switzerland, is an ideal additive for this purpose and should reduce initial setting times to as little as about 20 mins. SikaRapid-2 is also a hardening accelerator and also increases the early strength of the cement in the 6-24 hr period after coating.

In another embodiment a superplasticizer and/or hardening accelerator is added in addition to or instead of the setting accelerator to improve cement dispersion, and to improve the high early strength of the cement in (for example) the 8-24 hr period after coating, respectively. A suitable commercially available superplasticizer and hardening accelerator combination is the Sikament® - HE 200 concrete admixture product produced by Sika AG, Switzerland, which contains a blend of synthetic dispersing and complex-forming agents, namely sulphonated melamine-methanal condensate (melamine = triaminotriazine) and salts of thiocyanic acid.

In all embodiments, after the aggregate, cement, any additives and the water have been mixed, the coated aggregate mixture is poured onto a rotating or vibrating sieve while still wet. Alternatively, the mixture is poured onto a stack of multiple vibrating sieves with a gradually decreasing sieve opening size as one passes down the stack (e.g. 50 mm, 37.5 mm, 28 mm, 14 mm, 6.3 mm, 3.35 mm and 2.36 mm mesh size sieves). In each case, the coated aggregates which are retained on each (or the) sieve are directed towards or dropped onto a vibrating conveyor. Where a stack of sieves is used, there is provided one vibrating conveyor for each of the sieves of the stack of vibrating sieves. In this way, the coated aggregates are generally separated into different product lines with different coated particle size ranges, e.g. 3.35-6.3 mm, 6.3-14 mm, 14-28 mm, 28-37.5 mm and 37.5-50 mm which are the size ranges for coarse aggregate for road construction purposes specified in British Standard BS 4987 Part I, 1993. Fine coated aggregates of  $\leq 3.35$  mm coated

particles size (e.g.  $300\mu\text{m}$  -  $3.35\text{ mm}$ , and/or  $75\text{-}300\mu\text{m}$ ) can also be separated in the same way.

The coated aggregate will of course have a larger particle size than the original aggregate. In this way, aggregate of  $<3.35\text{ mm}$  particle size (eg  $2.36\text{ - }3.35\text{ mm}$ ) can be converted into coated aggregate of  $\geq 3.35\text{ mm}$  coated particle size and therefore will become suitable for use in road construction under BS 4987.

Instead of dropping the mixture onto a stack of sieves and then conveying separated fractions, it is also possible (though less preferable) to pour the whole mixture onto a single vibrating conveyor and separate the coated aggregates after conveying. In the present embodiment, however, the mixtures are separated by sieves initially, differently sized fractions conveyed separately (if necessary), and then the set or part-set coated aggregate re-sized after conveying in a second sieving stage. This second sieving stage also helps to reduce the number of "lumps", containing more than one coarse aggregate particle, which may have been formed.

It is noted that the vibrating and separating action of the sieves before conveying, as well as the vibrating action of the conveyor, helps to ensure that each particle of coated aggregate is separated from other particles, and that less "lumps", containing more than one coarse aggregate particle (i.e. of  $\geq 3.35\text{ mm}$  uncoated size), are formed.

The conveying time, from initial sieving to final deposit into a hopper, should be long enough to allow the coating of cement paste to reach its initial

setting either fully or partly before final deposit in the hopper. For example, if a setting accelerator (e.g. Sika®Rapid-2, described above) is used in the cement, then a conveying time of about 20 minutes should allow the cement coating to set sufficiently before deposit into a hopper. A longer conveying time, or deposit from the conveyor onto large drying sheets, will be necessary if OPC (drying time about 2-4 hrs) or rapid-hardening cement (drying time about 2-3 hours) are used without the above additives, to avoid excessive "lump" formation when part-set coated aggregate is deposited in a storage hopper.

After the initial setting time of the cement, a standard curing process is used, e.g. dropping the coated aggregate into water.

The dried coated aggregates can have a cement coating with an average thickness of 0.1-6 mm, but more usually the average thickness of the dried cement coating is 0.5-3 mm.

The coated aggregates have advantageous roughness, shear resistance (interlocking), bitumen adsorbence and deformation (rutting) resistance properties as disclosed in the following test results.

## **TEST RESULTS**

Some examples of tests made on the materials of the present invention are now given which demonstrate the potential advantages of the present cement coated aggregates over existing materials.

Some of the advantages of the coated aggregates are shown in the drawings attached hereto in which:

Figure 2 represents a comparison of the bitumen adsorption properties of coated and uncoated aggregate of a single particle size (2.36 mm before coating);

Figure 3 represents a comparison of the bitumen adsorption properties of graded uncoated aggregates and graded crushed coating material (75  $\mu\text{m}$  - 2.36 mm particle size);

Figure 4 is a direct shear and normal stress curve showing the interlocking properties of various tested aggregates (2.36 - 20 mm particle size before coating), as measured by the shear box method;

Fig. 5 is a graph showing percentage deformation vs. percentage of voids for coated and uncoated aggregates.

**Affinity between various asphalts and coated and uncoated aggregates**

The methods of testing chemically for the affinity between various asphalts and aggregates, as used in the USA Strategic Highways Research Programme (see Curtis reference 1.2 hereinabove), have been performed at Liverpool John Moores University on various premium and poor quality aggregates, both with and without the new coating to form a comparison.

The test method used (Curtis - ref 1.2 above) measures the initial adsorption of bitumen by the test materials at the beginning of each test. The test is an accelerated weathering test, during which bitumen is gradually desorbed from the material surface to generate a net bitumen adsorbency figure at the end of the test. This net bitumen adsorbence is the important figure as it includes the weathering and bitumen loss effects seen during the standard lifespan of road surfaces containing bitumen and aggregate.

For the tests, two aggregates were chosen: a crushed quarried rock "Arcow" and a smooth rounded land-won uncrushed natural gravel "Croxden". Both of these aggregates have very different surface properties but very similar density, aggregate impact value (AIV) and aggregate crushing value (ACV). Arcow represents high quality roadstone used in the pavement industry, whilst Croxden represents natural gravels currently considered unuseable.

Tests were performed firstly on finely graded samples of each aggregate and the coating material alone (which comprised ordinary Portland cement and which was checked to ensure that no aggregate material was

included), as shown in Fig. 3.

Further tests were then conducted on single sized samples (2.36 mm) of aggregate in its pure state and with the OPC coating applied as described hereinbefore: see Fig. 2.

In Figs. 2 and 3 it can be seen that adsorption of bitumen in Fig. 3 is nearly double the amount in Fig. 2. That is because the graded sample in Fig. 3 (75  $\mu$ m - 2.36 mm particle size) with many fine particles has a larger surface area available for adsorption. Graded samples are considered better for comparison than single sized samples because they represent the whole grading as found in any normal asphalt mixes.

Fig. 3 shows the ranking of performance of the two uncoated aggregates and the pure coating material tested. It can be seen that the uncoated aggregates in ascending order of chemical performance are as follows:- Croxden gravel has a lower net bitumen adsorbence (9.9%), while Arcow has a higher net bitumen adsorbence (29.7%). This ranking correlates with the performance expected if these aggregates were used to make road pavement. It can be seen that the net bitumen adsorption of the coating material is nearly 79% better than the best aggregate (Arcow) with a net adsorbence value of 53.1%.

The comparison of coated and uncoated aggregates, whose result is shown in Fig. 2, was performed on single sized particle samples (2.36 mm before coating, if any). A graded sample with particle sizes (before coating, if any) increasing from 2.36mm up to a maximum of 14mm was also considered.



The results in Fig. 2 show that when coating is applied to an aggregate, the net adsorption is improved by on average 110%. In this test the improvement in net bitumen adsorption for Croxden and Arcow was 93.8% and 125.3% respectively. This had the effect of bringing two different aggregates up to a similar level of bitumen/aggregate interaction performance.

The improvement evidenced in the above results shows that at both micro and macrostructure levels the coating shows significant improvement. This suggests that when aggregates are coated, their initial bond with bitumen (reflected in the initial adsorbence figures) and their resistance to stripping and ageing (reflected in the net adsorbence figures) is significantly enhanced because of the change in surface micro and macrostructure.

More importantly, bitumen adsorbence is enhanced because coating the aggregate with (lime-rich) cement makes the coated aggregate surface more oleophilic, i.e. more similar to a lime-rich (uncoated) aggregate, and hence more likely to bond well with bitumen.

#### **Roughness, friction (interlocking) and shear resistance**

At a macrostructure level, the cement coating changes the surface texture. The surface does become measurably rougher.

Table 1 shows roughness measurements of uncoated and coated Croxden and Arcow aggregates, as measured using a Uniscan optical surface profile instrument made by Uniscan Instruments, Buxton, UK. The aggregate particle size range used was 2.36 - 20 mm (before coating, if any). Coated

aggregates were prepared as described hereinabove.

**TABLE 1** Roughness of tested aggregates

Aggregate Type	Roughness ( $\mu\text{m}$ )	Average Roughness ( $\mu\text{m}$ )
<b>Uncoated Croxden</b>		
1	85.74	
2	82.74	85.7
3	89.38	
<b>Coated Croxden</b>		
1	124.75	
2	168.78	140.8
3	128.97	
<b>Uncoated Arcow</b>		
1	112.11	
2	117.05	111.3
3	104.75	
<b>Coated Arcow</b>		
1	276.22	
2	340.27	336.0
3	391.56	

In Table 1, for each of the four types of aggregate used, three samples were taken for roughness measurements, and an average value determined for each aggregate type. For Croxden (the natural uncrushed aggregate), the roughness changed from an average of 85.7  $\mu\text{m}$  (uncoated) to 140.8  $\mu\text{m}$  (coated), an improvement of 64%. Generally, coated natural uncrushed

aggregate has an average roughness in the range of 125-200  $\mu\text{m}$  or more usually in the range of 125 - 180  $\mu\text{m}$ .

The roughness of the crushed aggregate commonly used in pavement mixes (Arcow) increased from an average of 111.3  $\mu\text{m}$  (uncoated) to 336.0 (coated). Generally, coated crushed aggregate has an average roughness of 200 - 400  $\mu\text{m}$ . If fine aggregate (e.g. sand) is present in the coating, the average roughness is generally  $\geq 400 \mu\text{m}$  (not shown), because some of the fine aggregate will project from the surface of the coating, increasing the angularity of the coated aggregate.

Fig. 4 shows that the gap in the performance between the interlocking properties of crushed rock (e.g. Arcow) and rounded natural uncrushed gravel (e.g. Croxden) is bridged by coating the aggregates with cement as hereinbefore described. Fig. 4 shows the shear resistance properties, as measured by the shear box method, of graded aggregates with a particle size (before coating, if any) of 2.36-20 mm.

In Fig. 4, the coefficient of friction (angle of friction) of the coated aggregates improved by 47% (from  $11.9^\circ$  to  $17.48^\circ$ ) and by 21 % (from  $14.42^\circ$  to  $17.48^\circ$ ) compared to the uncoated aggregates, for the natural aggregate and crushed aggregate respectively. It is interesting to see that the friction coefficient lines of both coated materials coincide, i.e. that the interlocking and shearing resistance properties of the coated natural uncrushed aggregate and the coated crushed aggregate are substantially the same and are an improvement over the uncoated crushed aggregate (which already has

sufficient shearing resistance to be suitable for road use).

This improvement in the interlocking and shearing resistance of the modified aggregates leads to asphalt made of the coated aggregate exhibiting either the same stability of those mixes which contain crushed aggregate or an even better performance under the impact of both traffic and environmental conditions, as shown in Fig. 5.

Fig. 5 shows that both the coated natural uncrushed aggregate and coated crushed aggregate exhibit deformation (rutting) resistance properties either similar to or better than the most suitable crushed aggregate (Arcow) used in the UK. The deformation shown in Figure 5 is measured by Nottingham asphalt tester (NAT).

The above test results show that applying a cement paste, or cement paste containing strong particles, with or without applied additives, in accordance with the present invention, enables aggregates of low quality, such as natural (uncrushed), artificial, recycled and unsuitable crushed rock aggregates to be brought up to a recognised standard for use in pavement construction. It also enables aggregate (e.g. crushed aggregate) already of an acceptable quality to have its performance enhanced.

Coated aggregates can have improved strength, interlocking characteristics and durability. Mixes made with these types of aggregate become more durable, stable under the action of traffic loads and have longer design lives than those based on uncoated natural aggregates. The results from chemical tests and other laboratory tests such as repeat load axial, repeat load

indirect tensile, and wheel tracking tests show that coatings containing cement, such as Ordinary Portland Cement (OPC), are more stable and enable increased percentages of binder to be retained on the aggregates. Thus, the disadvantage of unacceptable aggregates which require less binder (which is not desirable as the finished product would quickly age and become brittle) is mitigated. Instead, the coated aggregates described herein have the advantages of reducing the ageing and long term brittleness of the final product.

The coated aggregates of the present invention are also more environmentally friendly, as described above.

CLAIMS

1. A particulate material suitable for use in construction comprising aggregate particles having a coating, wherein the coating comprises a set mixture of cement and water, and wherein the aggregate particles comprise natural uncrushed aggregate, recycled aggregate or artificial aggregate.

2. A particulate material as claimed in claim 1, wherein the cement comprises calcium, silicon, aluminium and oxygen in the form of one or more oxygen-containing compounds each containing one or more of calcium, silicon and aluminium.

3. A particulate material as claimed in claim 1 or 2, wherein the natural uncrushed aggregate comprises natural gravel.

4. A particulate material as claimed in any one of claims 1 to 3, including coated particles containing only one aggregate particle.

5. A particulate material as claimed in any one of claims 1 to 4, wherein the aggregate particles include those with a particle size of  $\geq 2.36$  mm (excluding the coating).

6. A particulate material as claimed in claim 5, wherein the aggregate particles include those with a particle size of 2.36-50 mm (excluding the coating).

7. A particulate material as claimed in claim 5, wherein the aggregate particles include those with a particle size of 2.36-14 mm (excluding the coating).

8. A particulate material as claimed in any one of the preceding

claims, wherein the aggregate particles substantially consist of those with a particle size as defined in any one of claims 5-7.

9. A particulate material as claimed in any one of the preceding claims, including that with a particle size of  $\geq 3.35$  mm.

10. A particulate material as claimed in claim 9, including that with a particle size of 3.35-50 mm.

11. A particulate material as claimed in any one of the preceding claims, substantially consisting of that with a particle size as defined in claim 9 or 10.

12. A particulate material as claimed in any one of the preceding claims, including that wherein the coating substantially completely surrounds the aggregate particles.

13. A particulate material as claimed in any one of the preceding claims, wherein the coating has an average thickness of 0.1-6 mm.

14. A particulate material as claimed in any one of the preceding claims, wherein the coating has an average thickness of 0.5-3 mm.

15. A particulate material as claimed in any one of the preceding claims, wherein the coating includes additives in addition to the cement, said additives comprising limes, microsilicas, and/or pulverised fuel ash (PFA).

16. A particulate material as claimed in any one of the preceding claims, wherein the coating has a total lime content of  $\geq 64\%$  of the total dry weight of the cement and additives in the coating.

17. A particulate material as claimed in any one of the preceding

claims, wherein the coating comprises a setting accelerator, a superplasticizer and/or a hardening accelerator.

18. A particulate material as claimed in claim 17, wherein the setting accelerator comprises a mixture of organic and inorganic complex-forming agents.

19. A particulate material as claimed in any one of the preceding claims, wherein the cement comprises Portland cement or rapid-hardening cement.

20. A particulate material as claimed in any one of the preceding claims, wherein the coating contains additional particulate matter.

21. A particulate material as claimed in claim 20, wherein the additional particulate matter has a smaller average particle size than that of the aggregate particles.

22. A particulate material as claimed in claim 20 or 21, wherein the additional particulate matter includes that with a particle size of  $75\ \mu\text{m}$  - 3.35 mm.

23. A particulate material as claimed in claim 20, 21 or 22, wherein the additional particulate matter comprises hard angular particles.

24. A particulate material as claimed in claim 23, wherein the hard angular particles comprise sand.

25. A particulate material as claimed in any one of claims 20 to 24, wherein some or all of the additional particles project from a surface of the coating.



26. A particulate material as claimed in any one of the preceding claims, including that with an average roughness of  $\geq 100 \mu\text{m}$ , as measured by optical surface profile.

27. A particulate material as claimed in any one of the preceding claims, including that with an average roughness of  $125 - 400 \mu\text{m}$ , as measured by optical profile.

28. A particulate material as claimed in any one of claims 20 to 25, including that with an average roughness of  $\geq 400 \mu\text{m}$ , as measured by optical surface profile.

29. A particulate material substantially as hereinbefore described and/or with reference to one or more of the accompanying drawings.

30. A method of making a particulate material as defined in any one of claims 1 to 29, comprising providing aggregate particles with a coating of cement paste, and allowing the cement coating to reach its initial setting.

31. A method as claimed in claim 30, wherein the coating is provided by mixing the aggregate particles with cement and water.

32. A method as claimed in claim 31, comprising mixing the aggregate particles with additional limes.

33. A method as claimed in claim 32, wherein 1-12 wt. % of additional limes by weight of dry cement powder used are added.

34. A method as claimed in claim 32, wherein 1-7 wt. % of additional limes by weight of dry cement powder used are added.

35. A method as claimed in any one of claims 31 to 34, comprising

mixing the aggregate particles with additional microsilicas and/or pulverised fuel ash (PFA).

36. A method as claimed in claim 35, wherein 1-12 wt. % of additional microsilicas and/or pulverised fuel ash (PFA) by weight of dry cement powder used are added.

37. A method as claimed in any one of claims 31 to 36, comprising mixing the aggregate particles with a setting accelerator, a superplasticizer and/or a hardening accelerator as defined in claim 17 or 18.

38. A method as claimed in any one of claims 31 to 37, comprising mixing the aggregate particles with additional particulate matter as defined in any one of claims 20 to 25.

39. A method as claimed in any one of claims 30 to 38, wherein the coated aggregate particles are separated before the cement coating is allowed substantially to reach its initial setting.

40. A method as claimed in claim 39, wherein the coated aggregate particles are separated by passing through or over one or more rotating and/or vibrating sieves.

41. A method as claimed in any one of claims 30 to 40, comprising disposing the coated aggregate particles onto a conveyor before the cement is allowed substantially to reach its initial setting.

42. A method as claimed in claim 41, wherein the conveyor vibrates in such a way as to aid separation of the coated aggregate particles from adjacent coated aggregate particles.

43. A method as claimed in any one of claims 41 to 42, comprising retaining the coated aggregate particles on the conveyor and/or an equivalent mechanism for a time sufficient to allow the coating to reach its initial setting at least partially.

44. A method as claimed in any one of claims 41 to 43, comprising adding dry cement and/or accelerating powder to the coated aggregate particles while they are still on the conveyor.

45. A method as claimed in any one of claims 41 to 44, comprising passing the coated aggregate particles through or over one or more sieves after conveying.

46. A method of making a particulate material substantially as hereinbefore described and/or with reference to one or more of the accompanying drawings.

47. A particulate material made by a method as defined in any one of claims 30 to 46.

48. A method of using a particulate material as defined in any one of claims 1 to 29 or claim 47, comprising mixing said particulate material with an asphalt and/or bitumen binder.

49. A material suitable for use in construction, comprising an asphalt and/or bitumen binder and a particulate material as defined in any one of claims 1 to 29 or claim 47.

50. The use in construction of a particulate material as defined in any one of claims 1 to 29 or 47, or a material as defined in claim 49.

51. The use of a particulate material as defined in any one of claims 1 to 29 or 47, or of a material as defined in claim 49, in construction of a surface.

52. The use as claimed in claim 51 as chippings or for surface dressing.

53. A method of constructing a surface comprising applying a material as defined in claim 49 to another surface.

54. A surface containing a particulate material as defined in any one of claims 1 to 29 or 47, or a material as defined in claim 49.

55. A use, method, or surface as claimed in any one of claims 51 to 54, wherein the surface comprises a road surface or an airfield surface.

56. A use, method, or surface as claimed in any one of claims 51 to 55, wherein the surface comprises flexible pavement.

57. A use, method, or surface as claimed in any one of claims 51 to 56, wherein the particulate material or material is included in the surfacing layers and/or roadbase layer of the surface.

58. An aggregate material of natural, artificial or crushed rock particles having a cement coating.

59. A method of modifying aggregate materials in order to improve their characteristics for use in pavement construction, wherein the aggregate material is provided with a coating of cement paste.

60. Materials, methods, uses and/or surfaces  
substantially as hereinbefore described in the example.

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Application No: GB 9818758.6  
Claims searched: 1 to 60

Examiner: Miss M M Kelman  
Date of search: 28 October 1998

## Patents Act 1977 Search Report under Section 17

### Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.P): C1A AVF2

Int CI (Ed.6): C04B 20/10

Other: ONLINE: WPI

### Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	WO 98/06677 A1 UNIVERSITÄT KARLSRUHE see the claims and page 13, line 20, to page 14, line 35	1,2,4,5,6, 7,8,9,10,1 1,12,13,14 ,15,17,19, 20,25,29,3 0,31,35,38 ,46,47,50, 54,58,59,6 0
X	US 4518431 A DUVIER see whole document	1,2,5,6,7, 8,9,10,11, 12,13,14,1 9,29,30,31 ,46,47,50, 54,58,59,6 0
X	US 4351670 A GRICE see column 1, line 60, to column 2, line 35, and column 4, line 52, to column 5, line 26, and the Figs.	1,2,4,5,6, 7,8,9,10,1 1,12,13,14 ,19,26,29, 30,31,39,4 0,43,46,47 ,50,54,58, 59,60

X Document indicating lack of novelty or inventive step  
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A Document indicating technological background and/or state of the art.  
P Document published on or after the declared priority date but before the filing date of this invention.  
E Patent document published on or after, but with priority date earlier than, the filing date of this application.



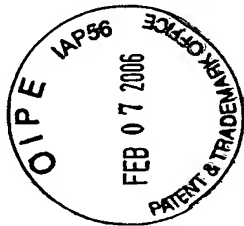
# The Patent Office

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Application No: GB 9818758.6  
Claims searched: 1 to 60

Examiner: Miss M M Kelman  
Date of search: 28 October 1998

Category	Identity of document and relevant passage	Relevant to claims
X	WPI Abstract Accession No. 94-188746[23] & JP 060127990 A (MITSUBISHI MATERIALS) 10 May 1994 see abstract	1,2,4,12,3 0,31,46,47 ,50,54,58, 59,60 at least



X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
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